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Evaluation of a New Microvolume 3HSc Electron Capture Detector and Ancillary Data System for Pesticide Residue Analysis

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EVALUATION OF A NEW MICROVOLUME ^{3}HSc ELECTRON CAPTURE DETECTOR AND ANCILLARY DATA SYSTEM FOR PESTICIDE RESIDUE ANALYSIS

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FOREWORD

The many benefits of our modern, developing, industrial society are accompanied by certain hazards. Careful assessment of the relative risk of existing and new man-made environmental hazards is necessary for the establishment of sound regulatory policy. These regulations serve to enhance the quality of our environment in order to promote the public health and welfare and the productive capacity of our Nation's population.

The Health Effects Research Laboratory, Research Triangle Park conducts a coordinated environmental health research program in toxicology, epidemiology, and clinical studies using human volunteer subjects. These studies address problems in air pollution, non-ionizing radiation, environmental carcinogenesis and the toxicology of pesticides as well as other chemical pollutants. The Laboratory develops and revises air quality criteria documents on pollutants for which national ambient air quality standards exist or are proposed, provides the data for registration of new pesticides or proposed suspension of those already in use, conducts research on hazardous and toxic materials, and is preparing the health basis for non-ionizing radiation standards. Direct support to the regulatory function of the Agency is provided in the form of expert testimony and preparation of affidavits as well as expert advice to the Administrator to assure the adequacy of health care and surveillance of persons having suffered imminent and substantial endangerment of their health.

This report represents a research effort to extend and improve analytical measurement of a variety of pesticides, polychlorinated biphenyls and other pollutants in our environment. The emphasis is on the evaluation of new detector systems for such environmental contaminants.

John H. Knelson, M.D.
Director,
Health Effects Research Laboratory

ABSTRACT

The performance of a linearized ^{3}HSc electron capture detector (ECD) and its ancillary data system was evaluated for use in the analysis of pesticide residues. Serial dilutions of pesticide standards were used to determine the maximum linear range and sensitivity of the detector. This detector was found to have a significantly greater linear range for the test compounds than a linearized ^{63}Ni electron capture detector evaluated. The sensitivity was only marginally better than the ^{63}Ni ECD.

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SECTION I

CONCLUSIONS

The micro-volume electron capture detector evaluated in this study was found to possess an extended linear range compared to another popular EC detector. In addition, the detector was a factor of ten more sensitive for certain compounds than another popular EC detector tested. These two advantages were more than nullified, however, by the susceptibility of the detector to contamination, which severely limited its useful life. The ancillary data system was useful in terms of data acquisition, storage, and computation of data, but the scope of application of the system is limited by certain design deficiencies.

SECTION II

INTRODUCTION

Since its first application in 1960 as a gas chromatographic detection technique,[1] the electron capture (EC) detector has played a major role in the analysis of pesticides, polychlorinated biphenyls, and other organic pollutants of environmental interest. The various types of EC detectors that are commercially available differ in the ionizing source used, the design of the detector cell, and the modes of electrical stimulation and measurement. Each of these facets can have considerable effect on the performance of an EC detector.

Three types of radioactive sources are commonly used in EC detectors: tritiated-titanium, nickel-63, and tritiated-scandium foils. The tritiated-titanium (^{3}HTi) sources are characterized by high specific activities which result in greater sensitivity and linear range than sources employing other radioactive isotopes. In addition, the low energy (18 keV) beta-rays emitted from such sources permit the construction of a detector with minimum internal volume, a characteristic which can be helpful in providing ionizing radiation throughout all of the interelectrode space. ^{3}HTi sources are prepared by occluding tritium in titanium-plated stainless steel or copper foils. Since tritium is easily lost from these foils, the maximum temperature at which they can be used is about 225°C. However, it is often necessary or desirable to operate an EC detector at higher temperatures; e.g., for analysis of high-boiling compounds and for "bake-out" (rejuvenation of

the foil by heating). The DC electrical stimulation typically employed for these sources interposes additional obstacles in achieving optimum detector performance. Electron capture detectors operated in the DC mode characteristically exhibit drifting baselines, sensitivity variations, and limited dynamic range.[2,3,4]

Detectors utilizing nickel-63 as the radioactive source can be operated at higher temperature (up to 375°C) but only at the expense of linear dynamic range and sensitivity. By operating the detector in the fixed-frequency pulsed mode, the limited linear range can be extended somewhat and the problem of baseline drift encountered with tritium detectors can be eliminated.[5,6]

Tritiated-scandium (^{3}HSc) sources have a maximum temperature limit of 325°C and source activities are available that approach one curie/cm^2. This provides a higher baseline current than any other electron-capture beta source. When this type of source is operated in the constant-current/variable-frequency pulsed mode, the linear range of the detector is significantly increased.[7]

The purpose of this study was to evaluate an electron capture detector employing a ^{3}HSc source operated in the constant current/variable-frequency pulsed mode. The ATC Model 140A detector, which is manufactured by Analog Technology Corporation, Pasadena, California, is the only high temperature tritium detector currently on the market. This detector is represented as having a maximum linear range of 10^6 when utilized in conjunction with an ATC Model 160A Memory and Display (MAD) system.

SECTION III

EXPERIMENTAL

In order to test the sensitivity and linearity of the detector against a wide range of response characteristics, a variety of compounds were chosen from several different chemical classes. Thirteen compounds or mixtures were selected which included chlorinated hydrocarbons, organophosphates, polychlorinated biphenyls and polychlorinated naphthalenes:

β-BHC	Ronnel
Hexachlorobenzene	Malathion
Aldrin	Ethyl Parathion
Perthane	Phorate
p,p'-DDE	Aroclor 1254
p,p'-DDT	Halowax 1014
Mirex	

All materials were primary reference standards obtained from the Pesticide Repository, Health Effects Research Laboratory, Research Triangle Park, N. C. Empirical formulae and chemical nomenclature are given for each standard in Table 1.

The test solutions of various concentrations were prepared from each standard in n-hexane (pesticide quality). Each of the standards consisted of a series of concentrations covering each decade from 1 pg/µl to 1 µg/µl. This provided a total of seven different concentration levels over which the linearity and sensitivity of the detector could be evaluated. The standards were evaluated individually to simplify

Table 1. COMPOUNDS USED FOR DETECTOR EVALUATION

Common Name	Empirical Formula	Chemical Name
β-BHC	$C_6H_6Cl_6$	β isomer of hexachlorocyclohexane
Hexachlorobenzene	C_6Cl_6	Hexachlorobenzene
Ronnel	$C_8H_8Cl_3O_3PS$	O,O-Dimethyl-O-(2,4,5-trichlorophenyl)phosphorothioate
Malathion	$C_{10}H_{19}O_6PS_2$	S-[1,2-bis(ethoxy carbonyl) ethyl]-O,O-dimethyl phosphorodithioate
Perthane	$C_{18}H_{20}Cl_2$	1,1-Dichloro-2,2-bis(p-ethyl phenyl)ethane
Aldrin	$C_{12}H_8Cl_6$	1,2,3,4,10,10-Hexachloro-1,4,4a,5,8,8a-hexahydro-1,4-endo-exo-5,8-dimethanonaphthalene
Ethyl Parathion	$C_{10}H_{14}NO_5PS$	O,O-Diethyl-O-p-nitrophenylphosphorothioate
Mirex	$C_{10}Cl_{12}$	Dodecachlorooctahydro-1,3,4-metheno-2H-cyclobuta[cd]-pentalene
p,p'-DDE	$C_{14}H_8Cl_4$	1,1-Dichloro-2,2-bis (p-chlorophenyl)ethylene
p,p'-DDT	$C_{14}H_9Cl_5$	1,1,1-Trichloro-2,2-bis (p-chlorophenyl)ethane
Aroclor 1254	$C_{12}H_5Cl_5$	mixture of polychlorinated biphenyls
Halowax 1014	$C_{10}H_{2-5}Cl_{5-5}$	mixture of polychlorinated naphthalenes
Phorate	$C_7H_{17}P_2PS_3$	O,O-Diethyl-5-[(ethylthio)-methyl]phosphorodithioate

identification and avoid potential coelution from the gas chromatograph.

The compounds were analyzed on a Tracor 560 gas chromatograph equipped with a 183 cm x 4 mm (i.d.) glass column packed with 3% OV-1 on 80- to 100-mesh Gas Chrom Q. The column was maintained at a temperature of 200°C with a 70 ml/min flow of 5% methane/95% argon carrier gas. The inlet ports and detector were maintained at 210°C and 240°C, respectively.

The Model 160A MAD system is designed to allow the collection of data in the form of automatically calculated peak area and peak heights, as well as simultaneous production of gas chromatograms on a conventional strip chart recorder. In addition, it has the capability of data retrieval from the disk file for later generation in the form of hard (paper) copy. Consequently, the data obtained in this study was reported from all four outputs when possible. Peak height was used as the response parameter for the data obtained from the strip chart chromatogram.

To ensure the statistical validity of the data, five replicate injections were made for each concentration level of each standard used in the study. The response data obtained was then normalized to a 5.0 µl injection volume to eliminate variation in response due to injection technique. The normalized data were averaged and the standard deviation and percent relative standard deviation for each

of the data outputs were calculated using a small sample statistics table.[8] The average response data for each of the output modes at each concentration level for a given standard were used for the final linearity calculations.

For a given standard, the linearity of response was calculated from the average response for each concentration level of each test solution. The calculation used in this analysis is:

$$\text{Normalized Response (K)} = \frac{\text{Response X Attenuation Factor}}{\text{Sample Amount}}$$

If a detector is linear, K should be constant within $\pm 5\%$ over the entire range of concentration levels. For automatically-calculated peak area and peak height data, the equation is reduced to:

$$K = \frac{\text{Response}}{\text{Sample Amount}}$$

since the attenuator control has no effect on these two forms of data output.

The sensitivity of the detector was established by injecting progressively smaller amounts of the standards into the GC system until the lower limit of detection(LLD) was reached. The LLD was defined as that amount of sample which would produce a signal equal to twice the noise level.

An aldrin standard with a concentration of 20 pg/µl was selected to serve as a reference to determine the long-term response stability of the detector. The standard was analyzed on a daily basis under identical

chromatographic conditions in order to detect any variation in response over the duration of the study. Some of the late-eluting compounds required more than one day of analysis time to complete the entire concentration series. In these cases, the data obtained from the analyses were adjusted in a corresponding fashion for any variations observed in the response from the aldrin standard. All data were corrected to the response level observed on the first day of analysis.

Since the baseline current available to an electron capture detector critically affects the overall system sensitivity and performance, this parameter was monitored on a daily basis. With the electrometer offset control centered and pure carrier gas flowing through the detector, the adjustable reference current was varied until a recorder output of zero volts was obtained. At this point the nominal baseline frequency was 5 kHz. The available baseline current was then recorded and the system was operated at that current level for the remainder of the day. Any subsequent variations in the baseline were corrected through use of the offset control.

SECTION IV

DISCUSSION

DETECTOR

As can be seen from the data presented in Table 2, the sensitivity of the detector was found to be comparable to that of a commercially-available linearized ^{63}Ni electron capture detector. Although the ^{3}HSc detector was more sensitive by a factor of 10 for three of the 13 standards tested, this was not considered to be a significant advantage over other EC detectors.

Table 2. COMPARISON OF THE LOWER LIMITS OF DETECTION (LLD) FOR TWO LINEARIZED ELECTRON CAPTURE DETECTORS

Compound	LLD (pg) ATC Model 140A	Tracor Linear ^{63}Ni
β-BHC	5	5
HCB	0.05	0.05
Ronnel	0.5	5
Malathion	50	5
Perthane	50	50
Aldrin	0.5	5
Ethyl Parathion	5	5
Mirex	5	5
p,p'-DDE	0.5	5
p,p'-DDT	5	5
Aroclor 1254	50	50
Halowax 1014	50	50
Phorate	50	50

The results of the linearity determinations are found in Tables 3 and 4. The detector has been represented[9] as having a linear range of 10^6. The results listed in Table 3 indicate that the maximum linear range achieved in this laboratory was 10^4 with a $\pm$5% deviation allowance. Linearity was not measurably greater when the response factor was allowed to vary by $\pm$10%. As can be seen from the tabulated data, the observed linear range was highly dependent on compound type.

This behavior has been previously reported for constant-current EC detectors which utilize a low-frequency pulsed operation.[10] The high-frequency operation employed in the ATC Model 140A should have circumvented this difficulty but apparently did not. Despite these aberrations, the linear range of the ^{3}HSc detector was found to be significantly greater than that of a Tracor linearized ^{63}Ni EC with which it was simultaneously compared.

Background noise from the detector was monitored on a daily basis in order to establish the maximum usable sensitivity of the instrument. The short-term noise characteristics of the detector when operated at maximum sensitivity were quite good. The amplitude of the short-term noise was usually less than 0.5% full-scale deflection (f.s.d.). With the exception of the four-minute period immediately following injection, the long-term noise was typically less than 2% f.s.d. when the detector was operated at maximum sensitivity. At the most sensitive attenuation, the chromatograms exhibited a 10% overshoot after the solvent peak which usually required approximately four minutes of recovery time to return

Table 3. MAXIMUM LINEAR RANGE WITH ±5% K-VALUE VARIATION

Compound	Linearity as calculated from: MAD Peak Area	MAD Peak Height	MAD Recorder Peak Height	Recorder Peak Height
β-BHC	10^3	10^3	10^4	10^4
HCB	10^2	10^1	10^2	10^2
Ronnel	$<10^1$	10^2	10^1	10^2
Malathion	10^1	10^1	10^1	$<10^1$
Perthane	10^1	$<10^1$	10^1	10^2
Aldrin	10^4	10^3	10^4	10^3
E. Parathion	10^1	10^1	10^2	10^1
Mirex	10^1	$<10^1$	10^2	$<10^1$
p,p'-DDE	10^2	10^1	$<10^1$	$<10^1$
p,p'-DDT	$<10^1$	$<10^1$	$<10^1$	$<10^1$
Aroclor 1254	10^1	10^2	10^2	10^2
Halowax 1014	$<10^1$	$<10^1$	10^1	10^1
Phorate	10^3	10^1	10^1	10^1

Table 4. MAXIMUM LINEAR RANGE WITH ±10% K-VALUE VARIATION

Compound	MAD Peak Area	MAD Peak Height	MAD Recorder Peak Height	Recorder Peak Height
β-BHC	10^4	10^3	10^4	10^4
HCB	10^2	10^2	10^2	10^2
Ronnel	10^3	10^3	10^3	10^3
Malathion	10^1	10^1	10^1	$<10^1$
Perthane	10^2	10^1	10^1	10^2
Aldrin	10^4	10^4	10^4	10^4
E. Parathion	10^2	10^2	10^2	10^1
Mirex	10^1	10^2	10^2	10^2
p,p'-DDE	10^3	10^3	10^1	10^1
p,p'-DDT	10^2	10^3	10^3	10^1
Aroclor 1254	10^1	10^3	10^2	10^2
Halowax 1014	$<10^1$	$<10^1$	10^1	10^1
Phorate	10^3	10^2	10^2	10^2

to baseline. The negative peaks reported by Olds[11] were not observed in this evaluation.

As previously mentioned, the zero-volt baseline is a measure of the detector baseline current, and is therefore a useful parameter for monitoring the condition of the detector source foil, the carrier gas purity, air leaks into the system, and all other conditions that can result in reduced baseline current. The stability of the detector in terms of baseline current variations is illustrated in Figure 1. As indicated in the graph, it was necessary to change the first foil (A) after the sixteenth day of use. It should be noted that the actual time from the beginning of the evaluation to the point at which the baseline current had degenerated to an unacceptable level spanned a period of 42 days. A statistical analysis of the baseline current levels shows that for foil A the arithmetic mean, standard deviation, and percent relative standard deviation are 1.24×10^{-8} amps, 2.5×10^{-9} amps, and 20% respectively. For foil B, the same analysis results in the respective figures of 1.66×10^{-8} amps, 2.3×10^{-9} amps, and 14%. This foil was used for a total of 25 days out of 72. In either case the variance is unacceptable when compared to the baseline current variations of a linearized ^{63}Ni EC detector as shown in Figure 2.

It could be argued that the observed variations for the ATC 140A are the results of factors independent of the detector itself. However, when it is considered that the detector under study and the linearized ^{63}Ni ECD against which it was compared were both mounted on identical gas chromatographs employing identical columns, ferrules, and septa,

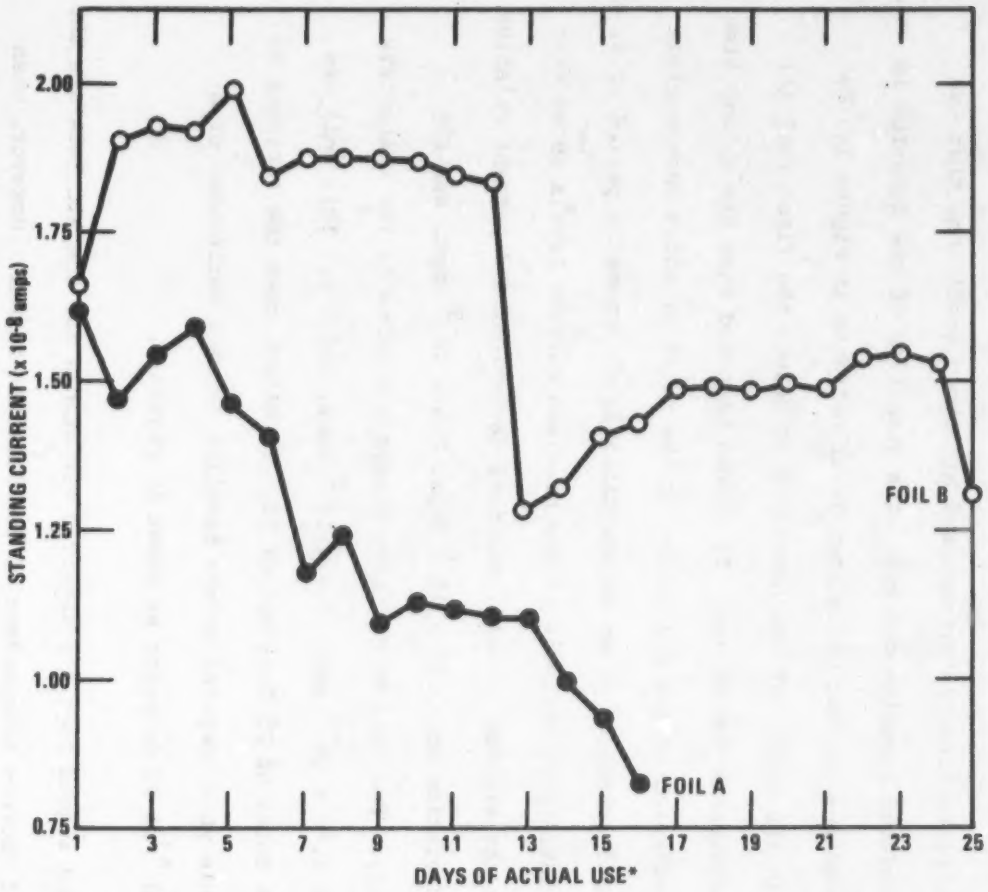

Figure 1. ATC 140 A baseline current levels. Currents monitored only on day of use. Total time Foil A in detector ca. 6 weeks; Foil B, ca. 14 weeks.

* Does not include weekends or other inoperative periods.

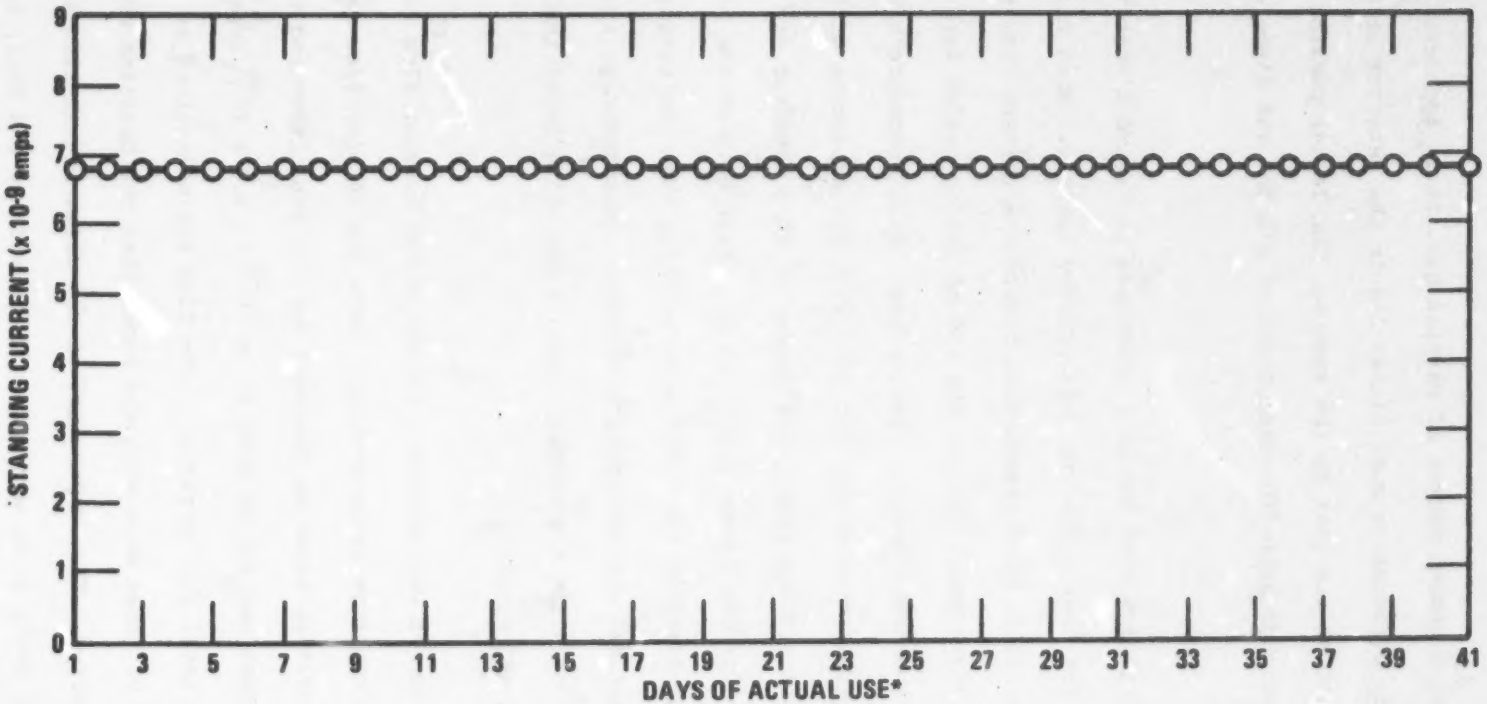

Figure 2. Linearized ^{63}Ni EC baseline current levels. Currents monitored only on day of use. Total time 20 weeks.

* Does not include weekends or other inoperative periods.

and utilizing a common source of carrier gas, it is apparent that the difference in performance must either lie in the detector or in the possibility of an air leak in the system. The latter possibility was eliminated through periodic inspections of all the gas lines and connectors.

It can be concluded from the data presented in Figure 1 that the overall lifetime of the source foil is unacceptably limited. When using most EC detectors, it is good practice to reduce the carrier flow rate to a minimum and add a purge flow at the end of the operating day in order to conserve the argon-methane carrier gas. This procedure was initially used in the evaluation of the ATC 140A with the exception of the application of a purge flow. The design of this detector did not include a purge line for the source cell. It was observed that the baseline current level steadily decreased with each day that the carrier flow rate was minimized for overnight operation. Consequently, it was necessary to maintain the carrier flow at operating level throughout the duration of the study.

On one occasion it was necessary to reduce the carrier flow rate overnight to conserve argon-methane. When the carrier flow was returned to normal operating level on the next day, it was discovered that the baseline current had fallen from 1.3×10^{-8} to 4.4×10^{-9} amps, an unacceptable level for operation. The flow was maintained at 70 ml/min for a period of three days with the hopes that the baseline current would be restored. The temperature of the detector was also increased from 240°C to 280°C in an attempt to decontaminate the foil, but this only resulted in further decrease of the baseline current.

In another instance, a regional electric power "brownout" required that all of the GC column ovens in the laboratory be shut down overnight. After power had been restored the following morning, the ATC detector required three days to return to its former baseline current despite the fact that the detector temperature and carrier flow rate had been maintained at normal operating levels. The ^{63}Ni EC detectors in the laboratory had returned to their former baseline current levels within an hour after power restoration.

The loss of baseline current, and consequently detector sensitivity in all cases cited above was probably caused by the adsorption of material eluted from the GC column onto one or both of the electrodes or by the coating of the electrodes with "dirt" or carbonaceous material. Such contamination can give rise to the formation of a contact potential that may either enhance or oppose the applied potential. The fact that the internal volume of the cell is only 180 microliters and that the detector has no provision for purging probably makes the ATC 140A more susceptible to contamination than other EC detectors.

The ATC Model 140A EC detector employs a scandium tritide source with an activity of one curie. Consequently, the safety aspects of operating and maintaining the device should be considered. The beta-particles emitted from the source have a maximum energy of 0.018 MeV and are absorbed by less than 1 mg/cm^2 of aluminum. As a result, there should be no detectable radiation from the tritium external to the detector

chamber. However, there is a possibility of tritium leakage from the cell through the exhaust port, especially at high temperature operation.

The installation kit provided with the detector includes several meters of flexible tubing which is used to vent the effluent from the exhaust port of the detector into a fume hood. Several of these detectors operated in an inadequately ventilated room could constitute a potential health hazard.

For laboratories that have in-house EC detector maintenance programs involving replacement of foils and cleaning of detector cells, additional safety considerations are necessary. When the foil of the ATC 140A EC detector employed in this evaluation was changed, wipe tests were made on various detector components during the disassembly process. One of the tests indicated low level contamination on the exterior surface of the detector housing. Another demonstrated that the outside surface of the detector cell itself was contaminated at the 0.5 μCi level. It could not be determined whether this high contamination resulted from tritium leakage from the source foil or from external contamination by the manufacturer during the assembly process. In either case, regular maintenance of this type of detector would require stringent observation of radiation safety practices.

MEMORY AND DISPLAY (MAD) SYSTEM

The Model 160A Memory and Display (MAD) system was evaluated in terms of display utility, storage and recall capability, ease of integration, and mechanical reliability. Data recorded in the MAD data memory is shown

graphically on the CRT display monitor with operator control of all display parameters including the memory segment displayed, attenuation, baseline offset, and linear or semilogarithmic presentation. The maximum number of data memory channels that are available in this system is 4096 (4K mode). The system is capable of splitting the memory into two equally-sized segments of 2048 channels each (2K mode). This capability offers two distinct advantages: first, when the CRT display is operated in the split-screen format two chromatograms that have been previously recorded in the 2K mode can be displayed simultaneously for comparison; secondly, a chromatogram capable of being contained within 2K of memory will only occupy half as much disk storage space as a chromatogram contained within 4K of memory. This latter characteristic becomes important when it is considered that the MAD system stores all available memory channels in the disk file whether or not data collection was terminated prior to filling all of the channels. Consequently, in order to conserve disk storage space, it is necessary to use the minimum allowable memory range. The frequency with which data points are collected can be controlled by the dwell time selector. With this control, the sampling interval can be varied from 0.01 seconds to 40.95 seconds in 0.01 second increments. Since the CRT is only capable of displaying 512 data points at any given time, depending on the selected display span and memory range, the display will show a maximum of every fourth data channel (2K mode) or every eight data channel (4K mode). The MAD system selects 512 equally-spaced channels for display if the displayed memory region contains more than 512 channels.

In this evaluation the display system was found to be quite useful to the extent that a chromatogram could be manipulated or attenuated after it was generated. The original chromatogram and any alterations thereof could be stored in the disk file memory for later retrieval or generated in hard copy form as a strip chart chromatogram or both. The primary difficulty encountered with the display system was the limited capability for real-time data acquisition. Table 5 lists the maximum data collection time as a function of dwell time for each of the memory ranges. In theory, these collection times should be adequate for most chromatographic applications. For example, at a dwell time of 40.95 sec/channel over a range of 4096 channels the system can collect data for over 46 hours. However, when actual chromatograms are generated while using the MAD system, it becomes apparent that the maximum useful dwell times and resulting data acquisition and display times are con-

Table 5. DATA COLLECTION TIME AS A FUNCTION OF DWELL TIME

Dwell Time (sec)	Maximum Data Collection Period (min)	
	2K Mode	4K Mode
0.01	0.34	0.68
0.1	3.4	6.8
1.0	34	68
10	340	680
40	1365	2730

siderably less than the theoretical limit. This is illustrated by the following case in which an aldrin standard was used to generate chromatograms for display on the MAD system. Initially the 2K memory range was used in conjunction with a display span of 2048 channels. Repeated injections of the aldrin standard at dwell times varying from 0.1 seconds to 3.0 seconds produced chromatograms which exhibited several interesting characteristics. It was found that a dwell time of 0.1 seconds was inadequate in that the display filled so rapidly there was insufficient time for the peak of interest to completely fit on the screen. In addition, as the dwell time was increased, the baseline noise increased correspondingly and increasing amounts of background had to be subtracted from the chromatogram to keep the baseline at roughly the same level from sample to sample. As the dwell time increased, it became more and more difficult to distinguish small sample peaks from the background noise. Additionally, the apparent position of the aldrin peak was shifted from right to left on the display as the dwell time was increased. While quantification by peak area measurement was not affected by this behavior, the peak height was found to increase proportionally with dwell time as expected.

The experiment was repeated using dwell times of 0.3, 10, and 40 seconds. Each dwell time was tested at span ranges of 2048, 1024, and 512. This was done in order to determine whether or not the GC peak of interest would be visible as it shifted to the extreme left side of the display at the higher dwell times. With the exception of the sample run at the 40-second dwell time, the peak was discernable at all span ranges. At

a dwell time of 40 seconds no peak was readily apparent at a span range of 2048. Neither the span range nor the dwell time had any affect on the peak area measurement. The measured peak height increased with increasing dwell time, but in a non-linear fashion. Again background noise increased appreciably as dwell time increased.

Using the results of these experiments, it was deduced that for most GC applications encountered in this laboratory the optimum dwell time is 0.3 seconds. This setting yields an acceptable compromise between maximum data collection time and acceptable background noise. However, this means that the chromatographic conditions of an experiment will have to be adjusted so that the compounds of interest will elute in less than 20.48 minutes, as this is the maximum allowable data collection period at this dwell time. Somewhat longer run times can be achieved by increasing the dwell time to a level required to collect the data while at the same time attenuating the display to the point where background noise is minimized. Unfortunately, this must be accomplished at the expense of sensitivity.

The MAD system exhibited another interesting characteristic during the course of the evaluation. When extremely dilute samples were being analyzed, the system sensitivity was not as good as that derived from data presented on a simultaneously-generated strip chart chromatogram. When using the MAD system, peak parameter computations are performed by manually defining the boundaries of a peak and allowing the system to automatically compute the peak area, amplitude, and retention time. The

peak is defined by setting movable upper and lower level cursors, which appear on the video display with the data. The area bounded by the data curve and the straight line joining the two cursors is automatically shaded by the MAD system so that the defined area is clearly indicated. An example of this can be seen in Figure 3. For single-component chromatograms, peak parameter computations were found to be no problem. However, in a multicomponent mixture, calculation of peak area and amplitude was tedious as each peak had to be defined manually prior to computation.

The storage and recall capability of the disk file is quite useful. As mentioned previously, data can be stored for later manipulation and calculation. During the course of the evaluation, no variations in a recalled data set in terms of peak limits, area, amplitude, and retention time were found. The major difficulty encountered with the memory system was the poor mechanical reliability of the disk file. The disk system had to be returned to the factory on three separate occasions for repairs. The MAD system can be operated without the disk unit but this severely limits the utility of the system.

Figure 3. Peak area integration on the ATC Model 160 Memory and Display (MAD) system.

SECTION V

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